

Determination of Tri-Saturated Glycerides in Lard, Hydrogenated Lard, and Tallow*

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THE only quantitative method described in the literature for determining tri-saturated glycerides in fat is that proposed by Hilditch and Lea (1). This method involves oxidation of the unsaturated glycerides in acetone solution with potassium permanganate and subsequent separation of the azelao-glycerides from the unchanged tri-saturated glycerides. Owing to the time-consuming and laborious nature of the manipulations involved, this method is not adaptable to routine analysis and has not been widely used.

In recent work (2) reported from this laboratory a fraction (first precipitate) was obtained from lard or tallow by crystallization that contained 90% or more of the total tri-saturated glycerides of the sample. This fraction also contained from 15 to 30% of di-saturated glycerides, which were almost entirely mono-oleins. From the fatty acid analysis it was possible to calculate the amount of tri- and di-saturated glycerides in the fraction.

Further investigation of the conditions of crystallization has led to the development of a fairly simple improved procedure for nearly quantitative determination of tri-saturated glycerides in lard, hydrogenated lard, and tallow. Since the precipitate fraction, which contains some di-saturated along with the tri-saturated glycerides, is reproducible, it may be useful as a constant in studies of factors that influence physical properties of fats. The method has not been applied to other fats and oils. However, it is not expected to be satisfactory for oils and fats that contain considerable amounts of saturated acids of low molecular weight.

Experimental

The two samples of lard (A and B) used were commercial steam-rendered products of good quality, consisting of 25% killing and 75% cutting fats. Lard B was caustic refined and filtered by conventional industrial methods. The hydrogenated lard samples² B-1, B-2, and B-3 represent successive stages of hydrogenation of Lard B. The hydrogenation was carried out in an industrial converter at 138-141° C. under 25-lb. hydrogen pressure. A concentration of 0.07 per cent Ni (Rufert flakes) was used as catalyst. The tallow was a good grade of edible kettle-rendered beef ruffle fat.

Analytical Characteristics and Fatty Acid Analysis

Iodine Value. A modification of the conventional Wijs method was employed for the "solid" glyceride fractions as follows: Five (5) mls. of standard Wijs solution are added to each 0.1-0.2 gram accurately weighed sample dissolved in 5 mls. of chloroform, and the solutions allowed to stand in the dark for 30 minutes. The excess reagent is titrated with 0.1 N thiosul-

fate, for which purpose a 10-ml. burette with 1/20 ml. subdivisions is used. Results by this modified method were in excellent agreement with those by the standard procedure. The standard Wijs method was used for the original fat samples.

Saponification Equivalent. A semi-macro procedure was satisfactory. To 0.14-0.17 g. of sample, accurately weighed, and contained in a 75-100 ml. round bottom flask (24/40), is pipetted 10 mls. of sodium propylate solution. The sodium propylate solution is made by adding enough cleanly cut sodium to propyl alcohol to make it 0.1 N. Then 2% of distilled water by volume is added. An air condenser fitted with a soda-lime tube is inserted in the flask, and the solution heated with a micro-burner to incipient boiling for 25 minutes. Under these conditions, rinsing the condenser is unnecessary. The neck of the flask is rinsed with 1 to 2 mls. of distilled water, and heating continued for an additional 2 minutes. A 10-ml. burette with 1/20 ml. subdivisions is used in titrating the excess alkali with 0.1 N HCl. Phenolphthalein (5 drops of 1% alcoholic solution) is satisfactory as an indicator if exposure of the sample to CO₂ is minimized. A blank containing only the sodium propylate solution is run similarly. The calculation is similar to that employed in the standard method. Values within $\pm 0.5\%$ of theoretical for pure methyl oleate and pure methyl stearate are obtainable with this procedure.

Thiocyanogen Values. The thiocyanogen values (T. V.) were determined with 0.1 N solutions of thiocyanogen, in accordance with the method described previously (3).

Spectrophotometric Analysis. A modification (4, 5) of the method described by Mitchell, Kraybill, and Zscheile (6) was employed for the determination of polyunsaturated fatty acids. The oleic acid content was calculated from the difference between total unsaturation as determined by the iodine value and that due to polyunsaturated components. The content of saturated acids was then obtained by difference.

Crystallization. The crystallizations were carried out in Erlenmeyer flasks of appropriate size, suspended in a constant temperature bath in such manner that the level of the acetone solution of fat was slightly below the level of the water in the bath. The temperature was maintained constant to within $\pm 0.3^\circ$ C. by a mercury thermoregulator and sensitive relay, which operated a pump for circulating cooling liquid through copper coils immersed in the bath. Uniformity of temperature in the bath was maintained by means of mechanical stirring.

Method for Determining Tri-Saturated Glycerides. Numerous experiments were carried out in which different conditions for crystallization of the fat samples were employed. The procedure chosen as best adapted to routine analysis and which involves the least time in man-hours is as follows: The sample of lard or tallow, 25-50 g., is dissolved in enough dry redistilled acetone (or analytical grade) to give a ratio of 40 mls. per gram of fat. The size of the sample can be

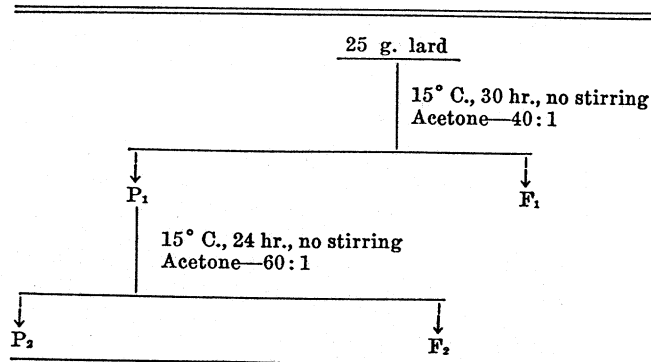
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adjusted according to the weight of "tri-saturated" precipitate expected; as a minimum, enough precipitate should be obtained to allow determinations of the iodine value and saponification value by semi-macro methods. Spectrophotometric analysis of the precipitate for polyunsaturated components is desirable but not always necessary. For lard, 50-g. samples will usually be adequate; for tallow, 25 g. may suffice.

CHART 1



The acetone solution of fat is placed in a constant temperature bath or cabinet regulated at 15° C. \pm 0.3° for 30 hours. The precipitate is filtered under moderate vacuum on a small weighed Büchner funnel (size 0 or 1), with No. 2 Whatman filter paper. The precipitate is pressed, and then washed with two 10-ml. portions of acetone cooled to 15° C., after which the suction is kept on for about 5 minutes. The funnel is reweighed to give a rough approximation of the amount of precipitate. Most of the precipitate is then transferred to another Erlenmeyer flask, and the remainder is quantitatively transferred with warm acetone. After the transfer sufficient acetone is added to give a ratio of 60 mls. per gram of weighed precipitate. The precipitate is dissolved by warming, and the solution allowed to stand about 24 hours at 15° C. The recrystallized precipitate is filtered, pressed, and washed as before and then transferred quantitatively to an accurately weighed 100-ml. round-bottom flask, with the aid of a little warm acetone. Most of the solvent is removed by distillation on a steam bath, the rest is removed under water pump vacuum and finally under high vacuum (oil pump) until constant weight of flask and contents is attained. The outline of the two crystallization steps is given in Chart 1.

Two methods for calculating the percentage by weight of tri-saturated glycerides may be employed: The first (I) consists in the determination of fatty acid composition from spectrophotometric data and iodine number and determination of saponification equivalent; the second (II) is a simplified approximation which requires only the iodine value, saponification equivalent, and weight of the recrystallized precipitate. These calculations can best be explained by citing a typical example:

Calculation I—Lard A, 25-g. sample

As shown in Tables 4 and 5, the recrystallized precipitate (a) has the following analytical characteristics:

0.7315 g = weight of fraction.

282.4 = saponification equivalent of the saturated acids (as glycerides), calculated by correcting the saponification equivalent of the fraction for the amount of unsaturated acids present.

295.1 = theoretical saponification equivalent of the unsaturated acids (as glycerides).

The fraction, as discussed later, is composed of tri-saturated and di-saturated glycerides. The proportion of each present in the fraction and the percentage of tri-saturated glycerides in the original sample are calculated as follows:

$$\frac{2(282.4)}{2(282.4) + 295.1} \times 100 = 65.65\% \text{ saturated acids (as glycerides) in the di-saturated glycerides present in the fraction.}$$

$$100 - 65.65 = 34.35\%, \text{ difference between theoretical percentage of saturated in tri-saturated and in di-saturated glycerides.}$$

*92.31 = percentage of saturated acids (as glycerides) found (Table 4).

$$100 - 92.31 = 7.69\%, \text{ difference between theoretical percentage of saturated in tri-saturated glycerides and the actual percentage in the fraction.}$$

$$\frac{7.69}{34.35} \times 0.7315 = 0.1637 \text{ g. wt. of di-saturated glycerides in the fraction.}$$

$$0.7315 - 0.1637 = 0.5678 \text{ g. wt. of tri-saturated glycerides in the fraction.}$$

$$\frac{0.5678}{25.0} \times 100 = 2.27\% \text{ of tri-saturated glycerides in Lard A.}$$

Calculation II—Lard A, 25-g. sample

The data in Table 4 show that oleic acid is the principal unsaturated acid in the fraction. It is apparent that the di-saturated glycerides present are principally mono-oleins and may be estimated approximately from the iodine number of the fraction, as follows:

$$7.02 = \text{I. V. of Determination a, Lard A, Table 4.}$$

30 = approximate I. V. of di-saturated mono-oleins, whose mean molecular weight is derived from the saponification equivalents as in Calculation I.

$$\frac{7.02}{30} \times 0.7315 = 0.1712 \text{ g. wt. of di-saturated glycerides in fraction.}$$

$$0.7315 - 0.1712 = 0.5603 \text{ g. wt. of tri-saturated glyceride fraction.}$$

$$\frac{0.5603}{25.0} \times 100 = 2.24 \text{ per cent tri-saturated glycerides in Lard A.}$$

* It is clear that the calculation of the amount of each type of glyceride present is based on the content of saturated acids (determined by difference) in the fraction. A direct determination of saturated acids would eliminate the necessity for determination of unsaturated acids. Unfortunately, however, available methods for determining saturated acids are cumbersome and of questionable precision, particularly when significant amounts of myristic acid or acids of lower molecular weight are present.

The results may also be expressed in percentages (mol), as in a previous paper (2).

Correction can be made for the solubility of tri-saturated glycerides in the volume of acetone employed. The solubility in acetone at 15° C. of tri-saturated glycerides from lard, obtained after repeated crystallization to remove all disaturated (mono-unsaturated), was 0.005 g. per 100 mls. of acetone. Since 1100 mls. of acetone (including washings) were used in the determination, the correction would be $11 \times .005 = .055$ g., which would increase the value for tri-saturated glycerides to 2.49% by Calculation I and 2.46% by Calculation II in the example cited. The solubility of the tri-saturated glycerides per 100 mls. of acetone at 10° and at 20° C., was 0.002 and 0.019 g., respectively.

Results and Discussion

Analytical characteristics and fatty acid composition of the lards, hydrogenated lard, and tallow, are given in Tables 1 and 2. The fatty acid composition calculated from the iodine and thiocyanogen numbers on the assumption that linoleic was the only polyunsaturated component was included only for the purpose of comparison with values obtained by the spectrophotometric method.

TABLE 1
Analytical Characteristics of Original Lard,
Hydrogenated Lard, and Tallow

Sample	I. V.	T. V.	Unsap. %	Wiley Melt. Pt. °C.
Lard—A.....	68.0	56.0	0.35	36.4
Lard—B.....	70.7	57.5	0.34	33.9
Hydrog. Lard—B ₁	60.3	54.4	0.34	44.5
Hydrog. Lard—B ₂	43.5	43.0	0.34	51.7
Hydrog. Lard—B ₃	25.9	25.7	0.34	58.6
Tallow.....	40.2	38.0	0.29	47.2

Examples of data obtained by crystallization of Lard A under different conditions are shown, Table 3. Consideration of the weights and iodine numbers of the recrystallized precipitates and corresponding filtrate fractions as well as the solubility of the tri-saturated glycerides (0.005 g. per 100 mls. of acetone at 15° C.) leads to the conclusion that essentially all the unsaturation of the final precipitate is due to di-saturated mono-unsaturated glycerides. This is particularly true for the recrystallized precipitates of low iodine values on which the calculations were made. Within limits, the conditions of crystallization may be varied appreciably without materially affecting the yield of tri-saturated glycerides. In general, however, the first crystallization should be carried out at temperatures not under 10° or over 15° C. Too low temperatures result in precipitation of large amounts of di-saturated glycerides unless excessive quantities of solvent are employed. Temperatures above 15° C. for the first crystallization result in low yields of tri-saturated glycerides. The conditions used in Experiment 5 (Table 3) were chosen as most practical for routine analysis, particularly when more than 2 or 3 samples are to be analyzed at a time. Conditions given in Experiment 3 permit more rapid determination but require additional equipment and a greater amount of time in manipulations.

Further information on the composition of the recrystallized precipitates given in Table 4 shows that the unsaturated glycerides in this fraction are composed principally of oleins, presumably di-saturated mono-oleins. The iodine value of the mono-oleins is approximately 30, as explained in Calculation II.

The glyceride composition of the hydrogenated samples of Lard B is somewhat less certain than that of

lard or tallow owing to the presence of "iso-oleic" acids formed during hydrogenation. As in the analysis of the original lard, some of the unsaturation of the precipitate fractions of B₁, B₂, and B₃ is probably due to di-saturated mono-oleins. It seems likely, however, owing to the greater unsaturation of these precipitates than those from Lard B, that an even greater proportion of it may be due to di-saturated mono-"isooleins" since this type of glyceride is formed during hydrogenation and would be expected to have physical properties closely akin to those of tri-saturated glycerides. The calculations (Tables 5 and 6) were made on the basis that the precipitate is composed of only tri-saturated and di-saturated glycerides (including di-saturated mono-"isoolein").

The possibility that small amounts of mono-saturated di-"isooleins" are present in the precipitate has been considered. Owing to this possibility the calculated percentage of tri-saturated glycerides for the hydrogenated samples may be in error, particularly those hydrogenated to a considerable extent, such as B-3. Theoretical calculations as well as some experimental evidence, however, have indicated that the total amount of mono-saturated di-"isooleins," even in a highly hydrogenated sample such as B-3, would be of the order of 1 to 2%. It is important, moreover, to note that the recrystallized precipitate obtained is a constant and reproducible fraction, and as such may be of value in correlation of factors that influence the behavior of shortenings during and after plasticizing.

The percentages of tri-saturated glycerides calculated by the two methods (I and II) are in good agreement, as shown by the data in Tables 5 and 6. The value for tallow, 14.2%, agrees well with the value, 14.7%, found in previous work (2), in which crystallizations were carried out on a kilogram of the same sample but under different conditions of crystallization. The values for tri-saturated glycerides given do not include corrections for solubility which, as explained in the calculations, would increase the values by about 0.22%.

Tri-saturated glycerides were also determined in Lard A by the acetone permanganate oxidation method of Hilditch and Lea (1). By this method 2.44 and 2.53% were obtained, as compared with 2.49 and 2.51% (corrected for solubility) by the crystallization method (Table 5).

The recovery of added tri-saturated glycerides was reasonably good by the crystallization method. When 0.5% was added to Lard A, the increase in tri-saturated glycerides found was 0.44%.

Although 25-g. samples of fat were used in this series of experiments, 50-g. samples are preferable for most lards because of the small amount of tri-saturated glycerides present. When trial 50-g. samples of

TABLE 2
Fatty Acid Composition of Original Lard, Hydrogenated Lard, and Tallow

Sample	Fatty Acid Composition							
	I. V.—T. V. Calculation			Calculated From Spectrophotometric Data				
	Linoleic	Oleic	Saturated	Linoleic	Linolenic	Arachidonic	Oleic	Saturated
	%	%	%	%	%	%	%	%
Lard—A.....	14.2	50.6	35.2	12.75	0.82	0.42	49.6	36.1
Lard—B.....	15.5	50.8	33.7	14.1	1.15	0.34	49.0	35.4
Hydrog. Lard—B ₁	6.8	56.5	36.7	6.80	0.35	0.08	56.2	37.2
Hydrog. Lard—B ₂	0.3	49.9	49.8	0.30	0.02	0.00	49.9	49.8
Hydrog. Lard—B ₃	0.0	29.9	70.1	0.10	0.00	0.00	29.9	70.0
Tallow.....	2.4	41.9	55.7	1.81	0.45	0.07	41.4	55.5

TABLE 3
Data on Fractions Obtained by Crystallization of Lard A (25 g.) Under Different Conditions

Expt. No.	Temp. of Cryst.	Solvent Ratio	Cryst. Time	Stirring	Precipitate			Filtrate			Tri- ² Saturated Glycerides
					No. ¹	Wt.	I. V.	No. ¹	Wt.	I. V.	
	°C.	mls./g.	hrs.			g.			g.		%
1.....	5	50	2	Mechan.	P ₁	4.2054	28.3	F ₁	20.7946	76.0
	10	400	2	Mechan.	P ₂	1.0645	14.2	F ₂	3.2343	37.4
	10	300	2	Mechan.	P ₃	0.7244	5.56	F ₃	0.3362	32.7	2.36
2.....	10	20	2	Mechan.	P ₁	3.6729	28.3	F ₁	21.3279	74.7
	15	30	2	Mechan.	P ₂	2.5559	23.3	F ₂	1.0942	42.5
	20	30	2	Mechan.	P ₃	1.2166	13.3	F ₃	1.3389	32.5
	20	30	2	Mechan.	P ₄	0.6920	5.77	F ₄	0.5097	24.0	2.24
3.....	10	50	2	Mechan.	P ₁	1.9742	23.3	F ₁	23.0461	71.7
	10	600	2	Mechan.	P ₂	0.7585	7.45	F ₂	1.2013	32.9	2.28
4.....	15	10	42	None	P ₁	2.7560	32.9	F ₁	22.2440	72.4
	15	40	42	None	P ₂	1.7048	21.1	F ₂	1.0420	52.6
5.....	15	40	30	None	P ₁	0.9063	12.3	F ₁	24.0375	70.3
	15	60	24	None	P ₂	0.6828	4.89	F ₂	0.2235	35.0	2.28
6.....	15	40	42	None	P ₁	0.8506	10.5	F ₁	24.1494	69.9
	15	60	24	None	P ₂	0.7024	5.17	F ₂	0.1482	35.6	2.33
7.....	15	40	42	None	P ₁	0.9900	14.3	F ₁	24.0100	70.2
	15	60	6	None	P ₂	0.7048	5.61	F ₂	0.2852	35.9	2.29

¹ The numbers P₂, P₃, and P₄ represent successive crystallizations of the first precipitate, P₁; likewise F₁, F₂, F₃, and F₄ represent the corresponding filtrate material obtained in those crystallizations.

² Calculation II was employed.

TABLE 4
Fatty Acid Composition of Solid Fractions Obtained by Crystallization of Lard and Hydrogenated Lard (25-g. samples) at 15° C.
Calculations Are Based on Spectrophotometric Data and Iodine Values.

Sample	Fraction	I. V.	Fatty Acid Composition				
			Lino-leic	Lino-lenic	Arachi-donic	Oleic	Saturated
	g.		%	%	%	%	%
Lard—A	a.....	0.7315	0.48	0.00	0.00	7.21	92.31
	b.....	0.7630	0.513	0.00	0.00	8.02	91.47
Lard—B	a.....	0.8870	0.512	0.001	0.00	6.75	92.74
	b.....	0.8856	0.512	0.001	0.00	9.21	90.29
Hydrog. Lard—B ₁	a.....	2.3500	0.293	0.00	0.00	18.79	80.92
	b.....	2.4980	0.230	0.00	0.00	19.74	80.03
Hydrog. Lard—B ₂	a.....	8.8330	0.00	0.00	0.00	23.23	76.77
	b.....	8.7000	0.00	0.00	0.00	22.66	77.34
Hydrog. Lard—B ₃	a.....	18.1700	0.00	0.00	0.00	17.32	82.68
	b.....	18.2500	0.00	0.00	0.00	16.97	83.03

TABLE 5
Glyceride Composition of Solid Fractions Obtained by Crystallization of Lard and Hydrogenated Lard at 15° C. ((25-g. samples).
Calculations Are Based on Fatty Acid Composition.

Sample	Fraction	Sapon. Equiv. of Fraction	Sapon. ¹ Equiv. of Sat. Acids as Glycerides	Glyceride Composition of Fraction				Tri-Saturated Glycerides in Original Sample	
				Tri-Saturated		Di-Saturated		%	Average %
	g.			g.	%	g.	%		
Lard—A	a.....	0.7315	283.3	0.5678	77.7	0.1637	22.3	2.27	2.28
	b.....	0.7630	283.6	0.5734	75.1	0.1896	24.9	2.29	
Lard—B	a.....	0.8870	284.5	0.6995	79.0	0.1875	21.0	2.80	2.67
	b.....	0.8856	284.5	0.6346	71.7	0.2510	28.3	2.54	
Hydrog. Lard—B ₁	a.....	2.3500	287.8	1.0680	45.4	1.2820	54.6	4.27	4.14
	b.....	2.4980	290.3	1.0210	40.9	1.4770	59.1	4.08	
Hydrog. Lard—B ₂	a.....	8.8330	291.0	3.0230	34.3	5.8100	65.7	12.10	11.71
	b.....	8.7000	291.6	2.8400	32.7	5.8600	67.3	11.32	
Hydrog. Lard—B ₃	a.....	18.1700	290.5	8.8300	48.6	9.3400	51.4	35.4	35.8
	b.....	18.2500	291.2	9.0500	49.6	9.2000	50.4	36.2	

¹ Calculated from the saponification equivalents of the fractions and their fatty acid compositions.

lard were used, the results were in excellent agreement with those reported for 25-g. samples.

The application of the method to other fats has not been investigated. It is expected to be reasonably satisfactory for fats whose saturated acids are principally acids of higher molecular weight, such as palmitic and stearic acids.

The method should prove of value in studies of hydrogenated fats, even though our lack of fundamental information concerning the glycerides of iso-

oleic acid lessens the quantitative aspects of results in such cases.

Acknowledgment

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Summary

Crystallization conditions are described which are suitable for obtaining almost quantitative precipita-

TABLE 6

Glyceride Composition of Solid Fractions Obtained by Crystallization of Lard, Hydrogenated Lard, and Tallow at 15° C. (25-g. samples). Calculations Are Based on Corrections for the Iodine Values of Di-Saturated Glycerides.

Sample	Fraction	Iodine Value of Fraction	Glyceride Composition of Fraction				Tri-Saturated Glycerides in Original Sample	
			Tri-Saturated		Di-Saturated		%	Average %
	g.		g.	%	g.	%		
Lard—A	a.....	0.7315	0.5603	76.6	0.1712	23.4	2.24	2.25
	b.....	0.7630	0.5648	74.0	0.1982	26.0	2.26	
Lard—B	a.....	0.8870	0.6889	77.7	0.1981	22.3	2.76	2.63
	b.....	0.8856	0.6258	70.7	0.2598	29.3	2.50	
Hydrog. Lard—B ₁	a.....	2.3500	1.0500	44.7	1.3000	55.3	4.20	4.27
	b.....	2.4980	1.0840	46.2	1.2660	53.8	4.34	
Hydrog. Lard—B ₂	a.....	8.8330	2.9740	33.7	5.8590	66.3	11.89	12.04
	b.....	8.7000	3.0460	35.0	5.6540	65.0	12.18	
Hydrog. Lard—B ₃	a.....	18.1700	9.1460	50.3	9.0240	49.7	36.58	37.02
	b.....	18.2500	9.3680	51.3	8.8820	48.7	37.47	
Tallow	a.....	4.7434	3.5481	74.8	1.1953	25.2	14.20	14.20
	b.....	4.7124	3.5500	75.3	1.1624	24.7	14.20	

tion of tri-saturated glycerides from lard, hydrogenated lard, and tallow.

Based on the weight and analysis of the precipitate obtained, a practical method for calculating the amount of tri-saturated glycerides in lard and tallow is proposed. When applied to several hydrogenated lards the method gave constant and reproducible fractions, which contained, in addition to tri-saturated glycerides, considerable amount of "isooleins." Some uncertainty is attached to the direct application of the method of calculation to these materials because of possible presence of mono-saturated-di-"isooleins."

Several samples of commercial lard examined contained about 2.5% of tri-saturated glycerides whereas

a sample of edible tallow contained about 14.0%. The proposed method, when applied to lard, gives results in good agreement with those obtained by the acetone-permanganate oxidation method of Hilditch and Lea. The chief advantage of the crystallization method is that much less time is required for the analysis.

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